PME RESEARCH LABORATORY, JULY 1980

PROJECT TITLE : ANALYTICAL INVESTIGATIONS

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## N-NITROSAMINES

### - Volatile Nitrosamines in SPOTLESS Cigarettes (1)

DMN and NPY in SS of 11 experimental cigarettes of the SPOTLESS series were determined. Nitrosamine values found and nitrate content of filler are summarized in Table 1. Although, because of some outstanding results, no final conclusions can be drawn, it can be seen from the figures in Table 1 that the elimination of nitrate (and water solubles) by aqueous extraction of blend 1 ØS-A-TOT and blend components 3 ØS-A-MD, 4 ØS-A-CH and 4 ØS-B-BU lowers the DMN and NPY levels in the sidestream smoke of cigarettes 2 ØS-A-TOT/SPO, 5 ØS-A-MD/SPO, 6 ØS-A-CH-SPO and 7 ØS-B-BU/SPO by 80 to 90 % (for codes of the experimental cigarettes see ref. 1).

### - Non-volatile Nitrosamines

We are not satisfied with the gas-chromatographic separation of non-volatile, tobacco-specific nitrosamines on GP 3 % SP-2100 DB/Supelcoport 100/120, the column we used so far for the quantitation of NNN, NAtB and NNK (2). Although showing acceptable resolution when synthetic nitrosamine mixtures are applied (fig. 1) under experimental conditions, e.g. when experimental samples are analyzed, a peak with RT 5.9 min (fig. 1) not separable from the NAtB peak falsifies the NAtB values. In addition, when operating under routine conditions, the life time of the SP-2100 column is limited.



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The separation of NNN, NAtB and of NNK could now be improved by using 5 % SP-2401 DB (Supelcoport 100/120) as stationary phase. This column not only gives a better separation of NNN and NAtB (fig. 3) but also a base line separation of the unknown peak (RT 10.46 min) from the NNN peak (fig. 4). The identification of the unknown peak (N-nitrosoanabasine ?) is presently under investigation.

SERVICE FOR OTHER GROUPS

- 80 tobacco lots (TLA) were analyzed for ISH.
- Potassium, calcium and magnesium were determined in 28 NINO extracts by atomic absorption.
- Nicotine was determined by gas chromatography in two NINO yeast samples (3) (4).

REFERENCES

(1) Compare F. Moser, PME Research Lab., Monthly Progress Report, June 1980, pp. 8-17.

(2) K.D. Brunnemann, PME Research Lab., Monthly Progress Report, December 1979, p. 5.

(3) Memo of P. Karbacher to F. Moser, July 4, 1980.

(4) Memo of F. Moser to P. Karbacher, July 8, 1980.

F. Moser



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Table 1 DMN and NPY (ng/c) *)**) in the sidestream smoke of SPOTLESS cigarettes.

| Cigarette Code | DMN (ng/c) | NPY (ng/c) | $N-NO_3$ (%) |
|---|---|---|---|
| 1 ØS-A-TOT | 267 | 58 | 0.27 |
| 2 ØS-A-TOT/SPO | 50 | 7 | 0.03 |
| 3 ØS-A-MD | 306 | 91 | 0.25 |
| 5 ØS-A-MD/SPO | 54 | 8 | 0.02 |
| 4 ØS-A-CH | 297 | 61 | 0.30 |
| 6 ØS-A-CH/SPO | 47 | 9 | 0.02 |
| 7 ØS-A/SPO/SPO | results pending | | -- |
| 1 ØS-B-TOT | results pending | | 0.35 |
| 2 ØS-B-TOT/SPO | results pending | | 0.015 |
| 3 ØS-B-FC | 191 | 77 | 0.03 |
| 6 ØS-B-FC/SPO | 128 | 15 | 0.007 |
| 4 ØS-B-BU | 413 | 177 | 0.46 |
| 7 ØS-B-BU/SPO | 94 | 18 | 0.05 |
| 5 ØS-B-OR | results pending | | 0.03 |
| 8 ØS-B-OR/SPO | results pending | | 0.002 |
| 9 ØS-B/SPO/SPO/SPO | 88 | 12 | -- |

*) Values as found; recovery approx. 80 %.

**) Mean values of 3 determinations.



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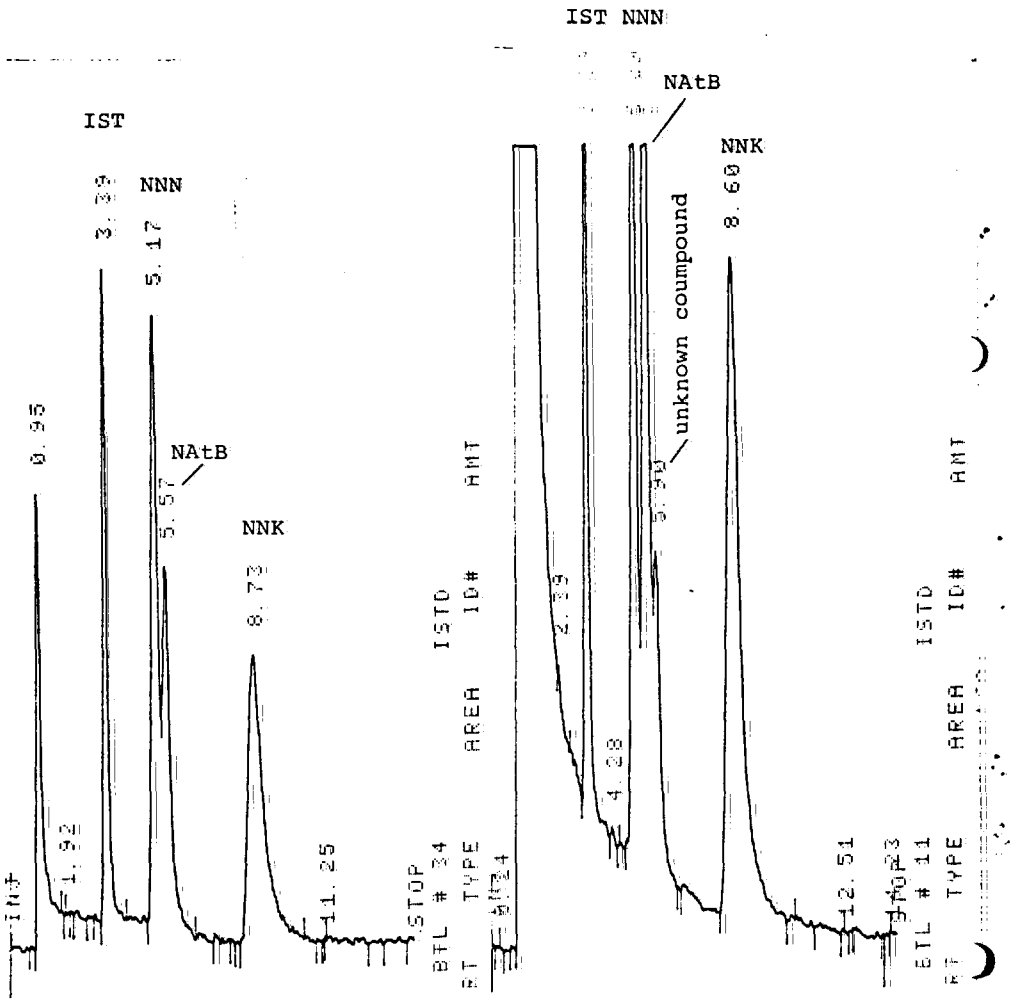
Gas-Chromatographic (GC/TEA) Separation of Tobacco Specific N-Nitrosamines on GP 3 % SP-2100 DB/Supelcoport 100-120 (3.5 m x 2.0 mm; oven temp. 213°)

Figure 1

Synthetic mixture of NNN, NAtB and NNK

Figure 2

Experimental sample (MS/Cambridge filter extract)



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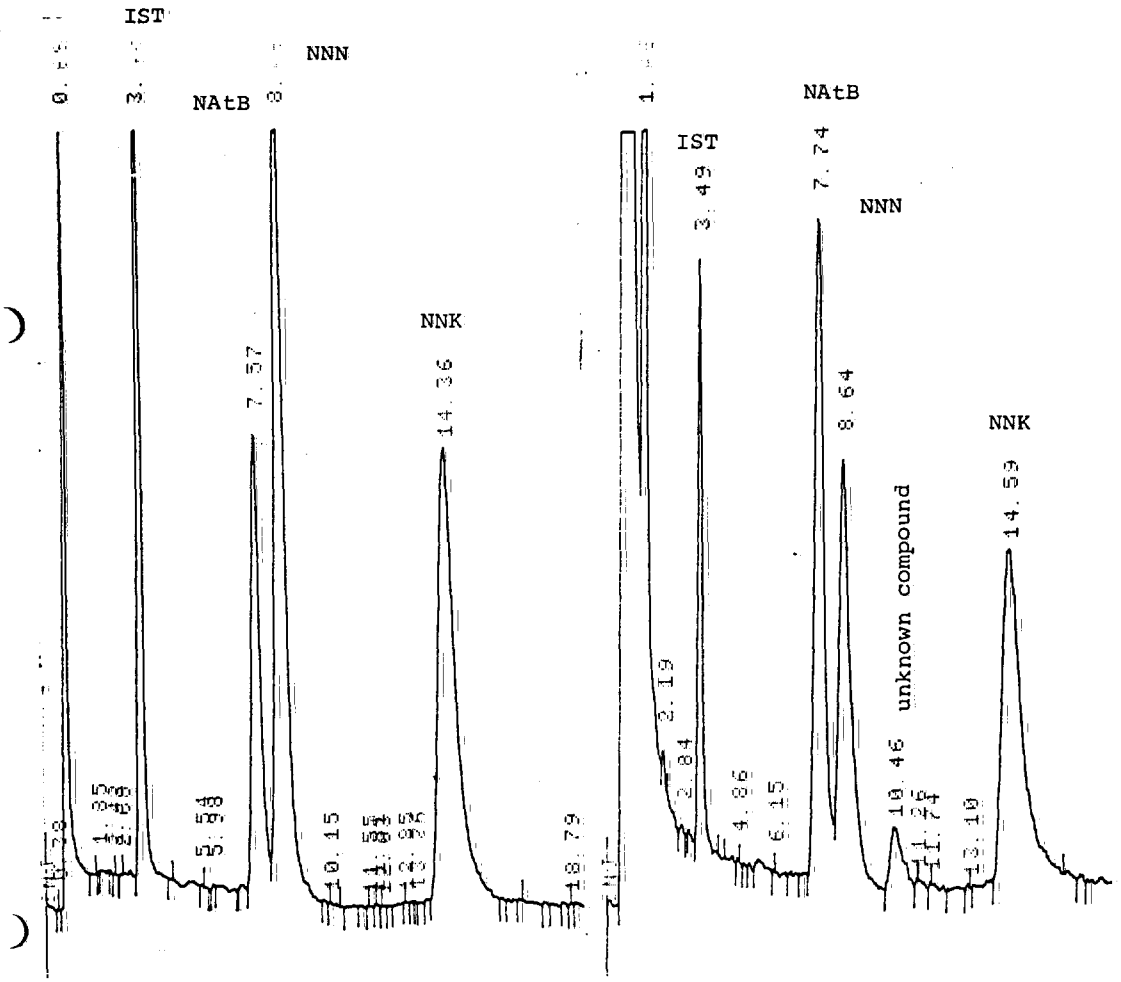
Gas-Chromatographic (GC/TEA) Separation of Tobacco Specific N-Nitrosamines on 5 % SP-2401 DB/Supelcoport 100-120 (3.5 m x 2 mm; oven temp. 220-250°).

Figure 3

Synthetic mixture of NNN, NAtB and NNK

Figure 4

Experimental sample (MS/Cambridge filter extract)



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